IRRADIATION TEST OF U-FREE NITRIDE FUEL AND PROGRESS OF PYROCHEMISTRY IN JAERI

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Abstract

Japan Atomic Energy Research Institute (JAERI) has proposed the double-strata fuel cycle for transmutation of long-lived minor actinides (MAs). The transmutation system is a Pb-Bi cooled sub-critical accelerator-driven system (ADS) with MAs nitride fuel. Nitride fuel has the advantage of accommodating various MAs with a wide range of composition besides superior thermal and neutronic properties. This paper concerns the status of the irradiation test of U-free nitride fuel and recent progress of pyrochemical process for nitride fuel in JAERI. Typical characteristics of nitride fuel for the irradiation test and the present schedule are described in addition to recent experimental results relating to pyrochemistry.

Introduction

Japan Atomic Energy Research Institute (JAERI) has proposed the double-strata fuel cycle for transmutation of long-lived minor actinides (MAs) such as Np, Am and Cm. [1] In this case, the partitioning and transmutation fuel cycle causes no negative impact on the commercial fuel cycle. The transmutation system proposed by JAERI is a Pb-Bi cooled sub-critical accelerator-driven system (ADS) with N-15 enriched MAs nitride fuel. Nitride fuel has the advantage of accommodating various MAs with a wide range of composition besides superior thermal and neutronic properties. Spent fuel from ADS is treated by pyrochemical process. Pyrochemical process has several advantages over wet process in treating such dedicated fuel for transmutation, including recycling feasibility of N-15 used in nitride fuel. [2]

In contrast to conventional fuel for power reactors, the dedicated fuel for transmutation is so-called U-free fuel that contains MAs as a primary component and non-fertile inert matrix as a diluting material. For example, JAERI has designed the 800 MWth-ADS in which (MAs,Pu)N diluted by ZrN is considered as a fuel material at initial loading. [3] However, information on such kind of fuel has been limited at present. So JAERI has started to examine the feasibility of fabrication and basic characteristics of the dedicated nitride fuel and also planned the irradiation test. [4] Development of nitride fuel and pyrochemical treatment of the spent fuel is the important subject for demonstrating feasibility of MAs transmutation cycle in addition to construction of high-energy proton accelerator and development of Pb-Bi technology.

This paper concerns the first irradiation test of U-free nitride fuel and recent progress of pyrochemical process for nitride fuel in JAERI following those presented in the last meeting at Madrid. [5] One He-bonded fuel pin incorporating PuN pellets containing ZrN and TiN was fabricated, followed by encapsulation for irradiation test at JMTR (Japan Materials Testing Reactor). The irradiation started in May 2002 and will go on for two years. Basic information on the irradiation behaviour of the dedicated fuel is expected from the following post irradiation examinations (PIEs). With regard to pyrochemical process, electrolysis of actinide nitride, electrode reaction of liquid metal electrode, recovery of actinides and nitride formation behaviour in liquid cadmium cathode have been investigated in a laboratory scale.

Transmutation fuel cycle

In the double-strata fuel cycle constituted by commercial power reactor fuel cycle (the first stratum) and transmutation fuel cycle (the second stratum), each fuel cycle shall be designed and optimised independently. Commercial power reactor fuel cycle insists on economy and effective utilisation of Pu in LWR or FBR, while transmutation fuel cycle focuses on effective transmutation of hazardous long-lived MAs and LLFP (Long Life Fission Products, such as ^{99}Tc and [129]I). A flowsheet of the second stratum proposed by JAERI is shown in Figure 1. Since the transmutation fuel cycle principally handles MAs partitioned from HLLW (High Level Liquid Waste) in the commercial fuel cycle, the heavy metal throughput of the second stratum is less than ~1/30 of that of the first stratum. [1] This point will result in compact equipment and effective MAs transmutation in the second stratum. According to JAERI's concept, one ADS plant transmutes MAs and LLFP produced by ten LWR plants of 1 000 MWe per year. In this case, the amount of MAs transmuted corresponds to ~250 kg/y and the MAs system inventory is estimated at ~5 500 kg based on some assumptions. Typical design parameters of ADS proposed by JAERI are shown in Table 1. [3] Fuel composition is N-15 enriched mononitride of 60%MAs+40%Pu diluted by inert matrices such as ZrN. In this case, however, the necessary amount of N-15 is only 20~30 kg/y if N-15 is recovered and recycled in the

second stratum, which will not be a penalty from economical viewpoint. This is one of the reasons that pyrochemical process for nitride fuel has been developed in JAERI for treating spent fuel for MAs transmutation.

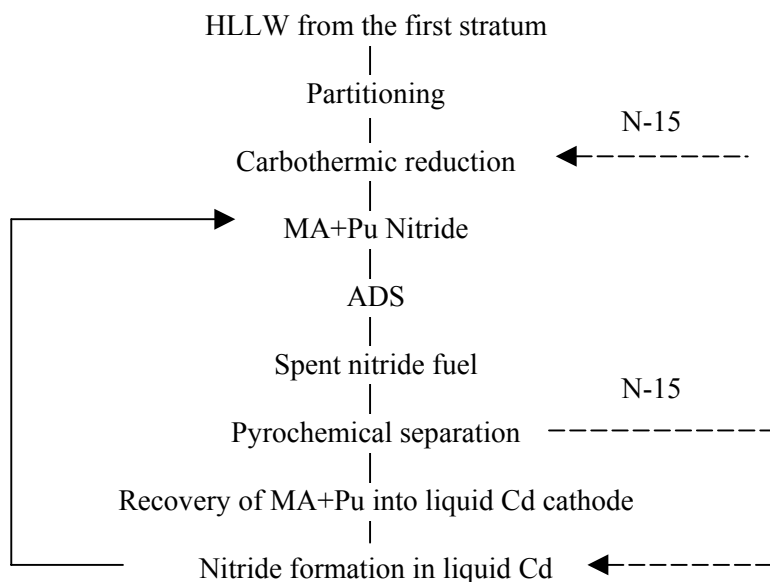Figure 1. **Flowsheet for MAs transmutation fuel cycle proposed by JAERI**

HLLW from the first stratum
|
Partitioning
| N-15
Carbothermic reduction ◄--------·
|
MA+Pu Nitride
|
ADS
|
Spent nitride fuel
| N-15
Pyrochemical separation --------┐
| |
Recovery of MA+Pu into liquid Cd cathode |
| |
Nitride formation in liquid Cd ◄--------┘

Table 1. **Typical design parameters of JAERI's ADS [3]**

Proton accelerator	1.5 GeV, 14 mA
Target material	Pb-Bi Eutectic
Coolant material	Pb-Bi Eutectic
Fuel	
Material[*,**]	(MA,Pu)N, ZrN
Inventory (MA/Pu)	2 500/1 660 kg
Thermal output	820 MW
k_{eff} (Initial/Max./Min.)	0.95 / 0.95 / 0.94
Coolant void reactivity	-4.8% dk/k
MA burnup	250 kg/year
Power density (Max./Av.)	310 / 180 MW/m^3
Coolant velocity (Max.)	2 m/s
Coolant temperature (Inlet/Outlet)	603 / 703 K

 * N-15 enriched nitrogen (99.9%), ZrN: Inert Matrix

 ** Pu added only at the initial loading

Irradiation test of nitride fuel

(U,Pu)N fuel

In JAERI, the irradiation test of nitride fuel was started in 1990 in order to demonstrate the performance as an advanced fuel for fast reactors. Up to now four He-bonded $(U_{0.8}Pu_{0.2})N$ fuel pins have been irradiated at JMTR and two $(U_{0.8}Pu_{0.2})N$ fuel pins at fast test reactor JOYO under joint research with JNC (Japan Nuclear Cycle Development Institute) as shown in Table 2. The results of PIEs for the nitride fuel irradiated at JMTR were described elsewhere [6,7] and those for irradiated at JOYO are preliminarily summarised as follows and to be published soon. 1) Any failure or defect of the fuel pin was not observed and good fuel performance was confirmed under the irradiation condition. 2) A higher swelling rate than MOX fuel was observed comparing at the similar smear density of fuel pins. Although the deformation of fuel pin itself was not severe, the indication of FCMI (Fuel-Cladding Mechanical Interaction) was seen at relatively low burn-up. It was also observed that He-gap width between pellet and cladding affected FCMI behaviour. 3) Even after irradiation at a higher linear power than conventional MOX fuel, as-fabricated fuel structure almost remained unchanged except for some porosity distribution. Fission gas release ratio was about a few percent at the most and segregation of solid fission products was not observed. 4) There was no distinct sign of FCCI (Fuel-Cladding Chemical Interaction) between nitride fuel and austenitic stainless steel cladding tube.

In addition, in May 2002 a new irradiation test was started at JMTR as mentioned before. One $(U_{0.85}Pu_{0.15})N$ fuel pin and one U-free nitride pin were encapsulated, whose characteristics are described in the following chapter. The schedule of this campaign is also included in Table 2 besides the irradiation tests performed so far.

Table 2. **Schedule of irradiation test of nitride fuel in JAERI**

Test No.	Reactor	Number of pins	Diameter of cladding (mm)	Linear power (kW/m)	Burn-up (GWd/t)	'91	'93	'95	'97	'99	'01	'03
1	JMTR	2	9.4	65	35	——	······					
2	JMTR	2	9.4	73	47	———		······				
3	JOYO*	2	8.5	78	40			———	——	——	··········	
4	JMTR	2**	9.4	40	(20)							——

*   Joint research with JNC   —— Irradiation   ·········· PIEs
**   One U-free nitride pin is irradiated besides one $(U_{0.85}Pu_{0.15})N$ pin.

Inert matrix nitride fuel

In the sub-critical core of ADS with U-free fuel, non-fertile inert matrix is usually added to the fuel as a diluting material. Essential criteria of the inert matrix are the stability against radiation damage (neutron, alpha and fission fragment), compatibility with the fuel for attaining high burn-up and thermal property not deteriorating characteristics of the fuel besides neutronic property. [8] Further, other important points lie in compatibility with fabricating and reprocessing technology of the fuel. There have been few results for nitride fuel containing inert matrix, so some design studies of

inert matrix fuel for ADS have been started for various kinds of fuel forms in JAERI. [9] The ratio of inert matrix in the fuel should be as low as possible from fuel cycle viewpoint, since most of the inert matrix in the spent fuel becomes waste unless proper recycling technology is actualised. In the experimental study, ZrN and TiN are chosen as candidates for inert matrix added to nitride fuel at first. Both ZrN and TiN have a face-centred cubic structure and high resistance to radiation damage is anticipated. Further, they have high melting temperature (2 973K for ZrN and 3 223K for TiN) and comparable thermal conductivity with nitride fuel although the reported data are rather scattered.

As a preliminary test PuN pellets containing ZrN and TiN were fabricated by conventional powder blending manner prior to fabrication of the irradiated sample. [4] PuN powder synthesised by carbothermic reduction of PuO_2 and those of ZrN and TiN powders obtained from CERAC Inc. were mixed in an agate mortar. The mixed powder was compacted into discs and heated in N_2-H_2 mixed gas stream at 1 673K for homogenisation. The discs were grounded and compacted into green pellet without any organic binder. Then the green pellets were sintered in Ar gas stream at 2003K, followed by heating in N_2-H_2 mixed gas stream for controlling stoichiometry. Although ZrN and TiN have the same structure of face-centred cubic as PuN, X-ray diffraction pattern of the products indicated a single-phase (Pu,Zr)N solid solution and a two-phase of PuN+TiN mixture, respectively. Single-phase (Pu,Zr)N pellets had a higher density than 90%T.D. (theoretical density), while two-phase PuN+TiN had a lower density than 80%T.D. So it was found that some consideration was necessary to attain high density that leads to structural stability for the latter.

Fuel capsule for irradiation test at JMTR

Following the preliminary test mentioned above, (Pu,Zr)N and PuN+TiN pellets for the irradiation test were fabricated by the almost similar manner. Since this is the first irradiation test of U-free nitride fuel in Japan, the design of the irradiation capsule became rather conservative. One He-bonded pin incorporating (Pu,Zr)N and PuN+TiN pellets and one $(U_{0.85}Pu_{0.15})$N fuel pin were encapsulated in a standard capsule of JMTR. Radial cross section of the 01-51A capsule is shown in Figure 2. In order to control the maximum temperature of Al-thermal medium below 673K, the maximum linear power was restricted at 40kW/m and Pu contents in (Pu,Zr)N and PuN+TiN pellets were chosen at 20wt.% for both cases. Any instruments were not provided except for neutron fluence monitors located outside thermal medium region.

Structure of the fuel pin incorporating (Pu,Zr)N and PuN+TiN pellets is shown in Figure 3. Respective fuel-stack length of (Pu,Zr)N and PuN+TiN pellets is 36 mm. Natural UN pellets were located both sides and between (Pu,Zr)N and PuN+TiN pellets as insulators. Diametric He-gap width between fuel pellets and cladding of austenitic steel was chosen at 0.15 mm following the previous irradiation test of (U,Pu)N. Large volume of gas plenum of atmospheric pressure was prepared for gas reservoir.

Typical characteristics of (Pu,Zr)N and PuN+TiN pellets for the irradiation test are shown in Table 3. Since the density of PuN+TiN pellets had to be increased, a ball-mill of WC was used for grounding the powder before compacting into green pellets. In this case the averaged sintered density of (Pu,Zr)N and PuN+TiN pellets was 90 and 87%T.D., respectively. On the other hand, atomic N/M (M=Pu+Zr or Pu+Ti) ratio corresponded to 1.01 and 0.76 for (Pu,Zr)N and PuN+TiN pellets, respectively, according to the results of chemical analysis. This means that (Pu,Zr)N solid solution has an almost stoichiometric composition, while PuN+TiN has some nitrogen vacancy, possibly for TiN phase. The lattice parameter of the (Pu,Zr)N solid solution almost agreed with the value assumed from the Vegard's law between PuN and ZrN, while those of PuN and TiN in PuN+TiN pellet remained unchanged from the respective compounds reported. Trace of Pu_2O_3 was identified in the diffraction pattern of PuN+TiN pellet.

Figure 2. **Radial cross-section of 01-51A capsule**
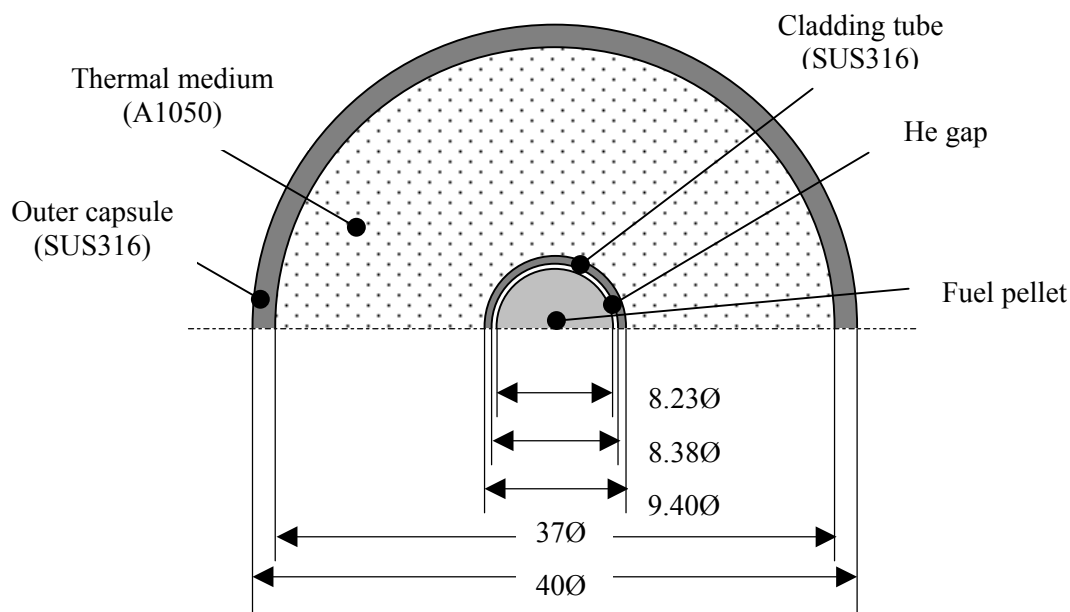

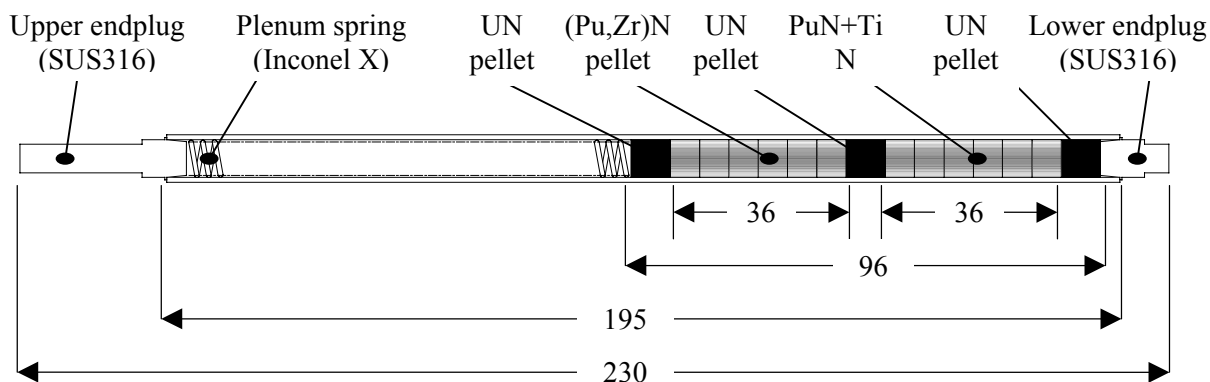Figure 3. **Structure of fuel pin containing (Pu,Zr)N and PuN+TiN pellets**

Table 3. **Typical characteristics of (Pu,Zr)N and PuN+TiN pellets for irradiation test**

Composition	Pu content (wt.%)[*]	Density (%TD)	Chemical analysis			X-ray diffraction	
			N (wt.%)	O (wt.%)	C (wt.%)	Phases	L.P.[**](pm)
0.10PuN+0.90ZrN	20	90	11.32	0.28	0.35	(Pu,Zr)N	460.9
0.06PuN+0.94TiN	20	87	14.42	0.79	0.50	PuN TiN	490.8 423.7

* nominal composition
** lattice parameter

Irradiation schedule

The irradiation of 01-51A capsule was started in May 2002. It will go on for two years over ten cycles at JMTR. After cooling for a few months, PIEs are to be carried out at RFEF (Reactor Fuel Examination Facility) of JAERI from the latter half of 2004. Temperature of the fuel pellets may be fairly low because of the conservative design mentioned above and high thermal conductivity of the fuel. Fuel central temperature at the first irradiation cycle was estimated at 1 253 and 1 153K for (Pu,Zr)N and PuN+TiN pellets, respectively, which were calculated from the linear power of 39 and 37 kW/m by use of GENGTC code. For the moment, however, thermal conductivities of the pellets only have been assumed by adding the thermal conductivity of each component reported in the literatures. The measurements of thermal conductivity of the pellets used in the irradiation test are planned near future. On the other hand, preliminary burn-up calculation indicates that the attained burnup after ten-cycle irradiation is 18 and 15GWd/t, about 16 and 20% of fission for initial Pu in (Pu,Zr)N and PuN+TiN pellets, respectively.

Pyrochemical technology

Anodic dissolution of nitride fuel

So far the electrolysis of UN, NpN, PuN and (U,Pu)N has been carried out in LiCl-KCl eutectic melts containing small amounts of trivalent actinide ions. Testing temperature of the electrolysis was chosen at 723, 773 and 823K. Cyclic voltammograms of mononitrides showed that oxidation to trivalent ions was considered as a rate determining step of electro-dissolution, since the current did not increase in proportion to square root of potential scanning rate and the cathodic peak seemed to shift more negative side as increasing scanning rate. In addition, the equilibrium potentials of UN, NpN and PuN vs. Ag/AgCl reference electrode (1wt.% AgCl) were determined from electromotive force measurements. The results at 723, 773 and 823K are summarised in Table 4. The equilibrium potential could be thermodynamically interpreted by comparison with the theoretical redox potential of the nitrides derived from free energies of formation and nitrogen partial pressure in the molten salt. [10] Further, the dissolution behaviour of lanthanide nitrides, which were surrogates of transplutonium element nitrides, in LiCl-KCl eutectic melts and the releasing behaviour of nitrogen gas were investigated by use of $CdCl_2$ as an oxidising agent. [11]

Table 4. **Equilibrium potential of UN, NpN and PuN vs. Ag/AgCl reference electrode determined from electromotive force measurements at 723, 773 and 823K**

	UN	NpN	PuN
723K	n.d.*	-0.779V	n.d.*
773K	-0.670V	-0.773V	-0.880V
823K	n.d.*	-0.766V	n.d.*

  * not determined

In case of the electrolysis of UN, however, the formation of ternary compound UNCl was identified at anode along with U^{3+} in some occasions. Since UNCl is hardly soluble in the molten salt, the formation of UNCl would adversely affect the efficiency of recovery at cathode. In order to study the anodic behaviour of UN in the chloride melt, stability diagrams (potential vs. log $p(N_2)$) of U-N-Cl system including UNCl were constructed from the thermochemical data of the compounds. On the

other hand, the formation of such a compound was not observed in case of the electrolysis of NpN and PuN. The results were derived from greater stability of trichloride of transuranium elements and lesser stability of the tetra valency compared with uranium. Nitride fuel for MAs transmutation does not contain U, so the problem of formation of actinide nitrogen chloride could be avoided in this case. [12,13]
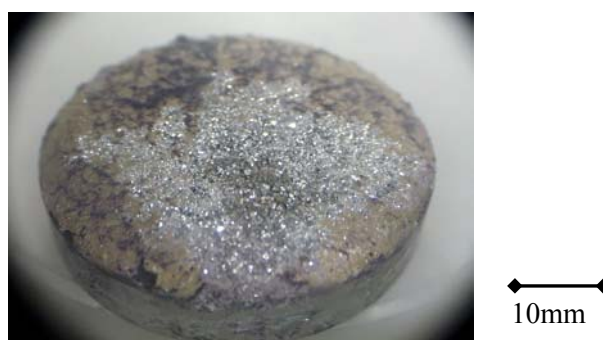
Recovery of Pu and U/Pu into liquid Cd cathode

Under the joint research of JAERI and CRIEPI (Central Research Institute of Electric Power Industry), an electro-refining apparatus equipped with liquid Cd cathode (LCC) was installed in a glovebox with high-purity Ar gas atmosphere. It was demonstrated from laboratory scale electro-refining in LiCl-KCl-PuCl$_3$ melt that Pu could be collected into LCC to ~10wt.% at high cathodic current efficiency without disturbance by solid phase formation. Further, metallurgical analysis showed that Pu collected beyond saturation in Cd phase formed an intermetallic compound PuCd$_6$ and precipitated at the bottom of LCC. [14]

Then co-recovering behaviour of U and Pu into LCC was investigated for developing pyrochemical reprocessing of fast reactor fuels. It is well known that U likely forms dendrite structure at the surface of the cathode and it often disturbs electro-refining process. So three electro-refining experiments were carried out at cathodic current density of 23.4mA/cm^2 in order to study the influence of the molten salt composition on the co-recovering behaviour. The U/Pu ratios in the LiCl-KCl-UCl$_3$-PuCl$_3$ molten were chosen at 1/12.2, 1/7.4 and 1/4.4. It was demonstrated that more than 10wt.% of U plus Pu was collected into LCC simultaneously at cathodic current efficiency higher than 87% in all cases. Further, no growth of dendrite structure was observed at the surface of LCC. On the other hand, metallurgical analysis showed that most of U and Pu collected beyond saturation in Cd phase formed PuCd$_6$ or PuCd$_{11}$-type intermetallic compound and precipitated at the bottom of LCC. Figure 5 shows the appearance of Cd ingot after electro-refining of the chloride molten salt with U/Pu ratio at 1/4.4. [15]

In this case the recovering behaviour of Am existing in Pu as impurity was also examined. It was observed that Am seemed to behave similarly as Pu during the experiment. Further, the separation factors of U/Pu and Am/Pu obtained by the present experiments almost agreed with those in LiCl-KCl/unsaturated liquid Cd system reported in the literature. [16] The experiments at higher cathodic current density are in progress for improving collection rate of actinides into LCC.

Figure 5. **Appearance of Cd ingot after electro-refining of the molten salt with U/Pu ratio at 1/4.4** [15]

10mm

Experiments on Am and Cm for pyrochemistry

For the moment, the basic electrochemical properties of Am and Cm necessary for development of pyrochemical process have not been well clarified. One of the reasons of lacking important data is that few facilities are available for the research on transplutonium elements in a controlled atmosphere. So a new facility, the Module for TRU High Temperature Chemistry (TRU-HITEC), is under construction at NUCEF (Nuclear Fuel Cycle Safety Engineering Facility) of JAERI Tokai. TRU-HITEC consists of three alpha/gamma cells shielded by steel and polyethylene and one glovebox maintained in high-purity Ar gas atmosphere. In TRU-HITEC, MAs such as ^{241}Am, ^{243}Am and ^{244}Cm are handled besides ^{237}Np, U, Pu and a small amount of spent fuel. [17]

One of the main research objectives in THU-HITEC is to investigate the pyrochemical process of nitride fuel for MAs transmutation. Two sets of molten salt electrochemical cells, a cathode processor, a furnace for nitride sample preparation and so on are to be installed in alpha/gamma hot cells. As the first Am-bearing experiment in TRU-HITEC, a basic study on pyrochemical process such as electrochemical behaviour of AmN in the chloride melt is considered. The ^{241}Am is recovered from aqueous waste produced at NUCEF and supplied to the experiments which will be started in 2003.

Conclusion

The present status of the irradiation test of U-free nitride fuel and recent progress of pyrochemical study in JAERI are summarised. One He-bonded fuel pin incorporating (Pu,Zr)N and PuN+TiN pellets was fabricated and subjected to capsule irradiation at JMTR. In this campaign ZrN and TiN were preliminary chosen as a testing material, but the selection of inert matrix and design study of dedicated fuel should proceed in accordance with fuel cycle strategy. Electro-refining of UN, NpN, PuN and (U,Pu)N have been carried out in LiCl-KCl eutectic melts at a laboratory scale, followed by recovery of actinides at solid or liquid cathode. Investigation of nitride formation behaviour of actinides in LCC and relating study on Am and Cm-bearing material are important subjects hereafter. TRU-HITEC will be a useful facility for the development of transmutation fuel cycle hereafter.

In JAERI, "High Intensity Proton Accelerator Project" is in progress under the joint programme with KEK (High Energy Accelerator Research Organisation), in which transmutation of MAs and LLFP is one of the important research subjects. Construction of proton linear accelerator (LINAC) was already started as the first phase and construction of Transmutation Experimental Facility (TEF) consisting of Transmutation Physics Experimental Facility (TEF-P) and ADS Target Test Facility (TEF-T) is planned as the second phase from 2006. [18] Keeping step with these progress of ADS technology in the physics and engineering aspects, experimental study on MAs nitride fuel and pyrochemical process of the spent fuel should further proceed for the development of transmutation fuel cycle.

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